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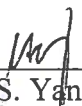
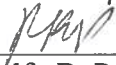
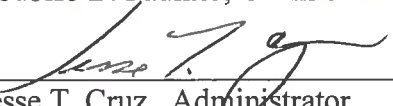
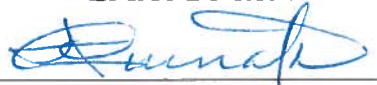
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Rev. no.

Ammonia-N, EPA 350.1
CH-01-03
10/31/2011
001

GUAM ENVIRONMENTAL PROTECTION AGENCY EMAS ANALYTICAL PROGRAM

STANDARD OPERATING PROCEDURE

DETERMINATION OF AMMONIA - N IN WATER BY FLOW INJECTION ANALYSIS COLORIMETRY

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Reviewed by:	 Jesse T. Cruz, Administrator EMAS Division	12/7/11 Date
Approved by:	 Ivan C. Quinata, Administrator Guam Environmental Protection Agency	12/13/11 Date

Periodic Review:
Signature

Title

Date



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1 SCOPE AND APPLICATION

- 1.1 This method covers the determination of ammonia-N in drinking, ground, and surface and saline waters, as well as domestic and industrial wastewaters.
- 1.2 The applicable range is 0.02 to 2 mg/L. The range can be extended for high level samples through dilution.
- 1.3 The quantitation limit for ammonia-N is 0.05 mg/L.

1.4 METHOD SUMMARY

- 2.1 Alkaline phenol and hypochlorite react with ammonia to form indophenol blue. The blue color formed is intensified with sodium nitroprusside which absorbs light at 630 nm. The absorbance is proportional to the concentration of ammonia in the sample.

3 INTERFERENCES

- 3.1 Calcium and magnesium ions may precipitate if present in sufficient concentration. EDTA is added to the sample in-line to prevent this problem.
- 3.2 Color, turbidity, and certain organic species may interfere. Turbidity is removed by manual filtration. Sample color may be corrected for by running the samples through the manifold without color formation.
- 3.3 Sulfide may interfere at levels greater than 2 mg H₂S/L. Samples containing concentrations greater than this should be diluted.
- 3.4 Salinity does not normally interfere in this method. The dissolved ion effect (salinity influence on absorbance) is less than 2%.

4 DEFINITIONS

- 4.1 Analytical Sample – Any sample in which ammonia is being determined, excluding standards, method blanks, or QC reference samples.



- 4.2 Calibration Blank (CB) – A volume of reagent water fortified with the same matrix as the calibration standards, but without the analyte.
- 4.3 Calibration Standard (CAL) – A solution prepared by diluting the primary stock standard solutions. The CAL solutions are used to calibrate the instrument response with respect to analyte concentration.
- 4.4 Field Reagent Blank (FRB) – An aliquot of reagent water or other blank matrix that is placed in a sample container in the laboratory and treated as a sample in all respects, including shipment to the sampling site, exposure to sampling site conditions, storage, preservation, and all analytical procedures. The purpose of the FRB is to determine if contamination is occurring in the field environment. Note: Field reagent blanks cannot be used for LD or LFM.
- 4.5 Field Duplicates (FD) – Two separate samples collected at the same time and place under identical circumstances and treated exactly the same throughout field and laboratory procedures. Analyses of field duplicates indicate the precision associated with the sample collection and storage as well as the laboratory procedures.
- 4.6 Instrument Performance Check (IPC) – A standard containing the analyte of interest that is used to verify the accuracy of analysis and monitor instrument drift. It is analyzed periodically through out an analysis sequence.
- 4.7 Calibration Verification (CV) solution: Initial (ICV) and Continuing Calibration Verification (CCV) solutions - A known value standard used to verify instrument performance during analysis. It is analyzed to verify that the initial calibration has not changed significantly during the analysis run. The CV fulfills the requirements of the IPC (4.6).
- 4.8 Laboratory Fortified Blank (LFB) – An aliquot of reagent water or other blank matrix to which known quantities of method analytes are added in the laboratory. The source of LFB must be independent of the calibration standards. LFB is analyzed like a sample, and its purpose is to determine whether the methodology is in control, and whether the laboratory is capable of making accurate and precise measurements. The LFB also fulfills the requirements of the QCS (4.14).
- 4.9 Laboratory Fortified Sample Matrix (LFM) – An aliquot of an analytical sample to which known quantities of the method analytes are added in the laboratory. The LFM is analyzed exactly like a sample, and its purpose is to determine whether the sample matrix contributes bias to the analytical results. The background concentration of the analytes in the sample matrix must be determined in a separate aliquot and the measured values in the LFM corrected for background concentrations.



- 4.10 Laboratory Duplicate (LD) – An aliquot of sample prepared and analyzed separately with identical procedures. Analysis of the sample and LD indicates precision associated with the laboratory procedures, but not with sample collection, preservation or storage procedures.
- 4.11 Laboratory Reagent Blank (LRB) – An aliquot of reagent water or other blank matrix that is treated exactly like a sample. The LRB is used to detect sample contamination resulting from the procedures used to prepare and analyze the samples in the laboratory environment.
- 4.12 Linear Calibration Range (LCR) – The concentration range over which the instrument response is linear.
- 4.13 Method Detection Limit (MDL) – The minimum concentration of an analyte that can be identified, measured and reported with 99% confidence that the analyte concentration is greater than zero.
- 4.14 Quality Control Sample (QCS) – A standard containing ammonia that is used to verify the accuracy of the analysis. The method requires that the source of the QCS must be independent of the calibration standards and that the QCS be analyzed quarterly.
- 4.15 Quantitation Limit (QL) – The concentration at which confidence in the reported value requires no qualifying remarks. The QL, also called as the practical quantitation limit (PQL) is about 5X the MDL and represents a practical and routinely achievable detection limit with a relatively good certainty that any reported value is reliable.
- 4.16 Stock Standard Solution (SSS) - A concentrated solution containing the method analyte prepared in the laboratory using assayed reference materials or purchased from a reputable commercial source.
- 4.17 Sample Delivery Group (SDG) – A group of twenty samples or less from the same case that is sent to the laboratory for analysis.

5 HEALTH AND SAFETY

- 5.1 The toxicity or carcinogenicity of each reagent used in this method has not been fully established. Each chemical should be regarded as a potential health hazard and exposure should be as low as reasonably achievable. Safety precautions must be taken when handling solutions and samples. Protective clothing including lab coats, safety glasses and gloves must always be worn. Contact lenses must not be worn. If



solutions come into contact with your skin, wash thoroughly with soap and water. Contact your Supervisor or Health and Safety Coordinator to determine if additional treatment is required.

5.2 The following chemicals have the potential to be highly toxic or hazardous, for detailed explanations consult MSDS.

5.2.1 Sodium Hydroxide

5.2.2 Phenol

5.2.3 Sulfuric Acid

6 SAMPLE HANDLING AND PRESERVATION

6.1 Samples should be collected in precleaned plastic or glass bottles. Volume collected should be sufficient to insure a representative sample, allow for replicate analysis and minimize waste disposal.

6.2 Samples may be stored frozen in polyethylene bottles or chemically preserved by adjusting to pH <2 with 2 ml of 8N H₂SO₄ per liter of sample and storing them at 4°C. Analyze samples within 28 days.

7 EQUIPMENT AND SUPPLIES

7.1 Lachat QuikChem 8000 Series FIA+ instrument, ammonia manifold and the Lachat QuikChem data system and software

7.2 Analytical Balance, capable of accurately weighing to the nearest 0.0001 g

7.3 Class "S" weights

7.4 Drying oven, capable of being controlled at 140 ± 5°C

7.5 Desiccator

7.6 Glassware – Class A volumetric flasks and pipettes or plastic containers as required.

7.7 PVC pump tubes must be used for this method.



8 REAGENTS AND STANDARDS

8.1 Preparation of Reagents

Use ASTM Type II reagent water for all solutions.

8.1.1 Reagent 1. Buffer Chelating Agent: In a 1 L volumetric flask, dissolve 50 g disodium ethylenediamine tetraacetate (Na_2EDTA) and 11 g sodium hydroxide (NaOH) in about 900 mL DI water. Stir to mix and dilute to the mark with reagent water. Prepare fresh monthly.

8.1.2 Reagent 2. Phenate Reagent:

CAUTION: Wear gloves. Phenol causes skin burns and is rapidly absorbed into the body through the skin. Do not degas this reagent.

In a 1 L volumetric flask, dissolve 83 g crystalline phenol ($\text{C}_6\text{H}_5\text{OH}$) (or 94 mL liquid phenol) in approximately 500 mL reagent water. While stirring, slowly add 32 g sodium hydroxide (NaOH). Cool, dilute to the mark with reagent water, and invert to mix. The color of this reagent darkens with age, increasing the baseline absorbance. Prepare fresh reagent after 72 hours. Prepare fresh every 3 – 5 days and discard when turns dark brown.

8.1.3 Reagent 3. Sodium Hypochlorite: Dilute 250 mL sodium hypochlorite (4-6% NaOCl) to 500 mL with reagent water. Prepare fresh daily.

8.1.4 Reagent 4. Sodium Nitroprusside: Dissolve 1.75 g sodium nitroprusside in 500 mL reagent water. Prepare fresh every 1 – 2 weeks.

8.1.5 Reagent Water – carrier

8.2 Preparation of Standards

8.2.1 **Stock Standards:** Stock standard solutions may be purchased as certified solutions or prepared from ACS grade materials (dried at 100°C) and are stable for six months when stored at 4°C .

8.2.2 **Standard 1. Stock Standard 1000 mg/L Ammonia-N:** In a 500-mL volumetric flask, dissolve 1.909 g of primary standard grade anhydrous ammonium chloride



(NH_4Cl) that has been dried for 2 hours at 100°C in about 200 mL reagent water, dilute to the mark and invert to mix. Prepare fresh every six months. Alternatively, a commercially prepared standard can be obtained from a reputable supplier.

- 8.2.3 **Working Standards Ammonia-N** – Prepare fresh daily. The following five calibration standards, QL and CV solutions are prepared using Standard 1 (8.2.2), 1000 mg/L Ammonia-N and diluted with reagent water.

Calibration Standard	Volume of Standard 1	Final volume
1.00 mg/L	100 uL	100 mL
0.50 mg/L, CV	50 uL	100 mL
0.25 mg/L	25 uL	100 mL
0.10 mg/L	10 uL	100 mL
0.05 mg/L, QL	10 uL	200 mL
Blank	0 uL	100 mL

QCS (or LFB): 0.50 mg/L --- must be prepared from a second source stock standard.

9 QUALITY CONTROL PROCEDURES

- 9.1 Guam EPA operates a formal quality control (QC) program. The QC program consists of an initial demonstration of laboratory capability, and the periodic analysis of laboratory reagent blanks, fortified blanks, QCS samples and other laboratory solutions as a continuing check on performance. The laboratory is required to maintain performance records that define the quality of the data that are generated.
- 9.2.1 Initial Demonstration Proficiency – Each analyst must complete an initial demonstration of proficiency prior to analyzing samples following this method.
- 9.2.3 MDL – A method detection limit must be confirmed annually and must be $<1/2$ the QL or corrective action must be initiated.
- 9.2.4 QCS – a QCS must be prepared and analyzed when beginning the use of this method, on a quarterly basis or as required to meet data-quality needs. The source of the QCS must be independent of the calibration standards. The QCS verifies the calibration standards. Guam EPA Laboratory fulfills the requirements of the QCS with analysis of the LFB.
- 9.3 Routine Analytical Quality Control



- 9.3.1 The instrument must be calibrated with a blank and 5 standards. The correlation coefficient of the calibration curve must be ≥ 0.995 or the instrument must be recalibrated.
- 9.3.2 CV – The accuracy and stability of the calibration shall be verified by the periodic analysis of a CV standard. It must be analyzed at the beginning of an analytical run (the ICV), after every 10 analytical samples (the CCV), and at the end of an analytical run (the closing CCV). The CV solution should be prepared from the same standard stock solutions used to prepare the calibration standards.

The recovery of ammonia in the CV is calculated as follows:

$$\% R = \frac{M}{T} \times 100$$

Where

%R = percent recovery of the standard

M = measured concentration of ammonia-N, mg/L

T = true concentration of ammonia-N in the CV, mg/L

If the CV recovery exceeds the limits of 90 – 110%, the analysis shall be terminated. The cause of the poor recovery must be determined and the problem corrected. The instrument must be re-calibrated and all samples not bracketed by acceptable CV results must be reanalyzed.

- 9.3.3 CB (ICB/CCB) – The stability of the baseline must be monitored by analyzing a CB immediately after every CV standard. If the absolute value of the CB result equals or exceeds the QL, the analysis must be terminated. The cause of high CB result must be determined and the problem corrected. The instrument must be re-calibrated and all samples not bracketed by acceptable CB results must be reanalyzed.
- 9.3.4 QL – The accuracy of the calibration at the reporting limit shall be verified by the analysis of a QL standard. The QL must be analyzed at the beginning of each analytical run, prior to the analysis of environmental samples. The recovery of ammonia in the QL is calculated as follows:

$$\% R = \frac{M}{T} \times 100$$

Where

%R = percent recovery of the standard

M = measured concentration of ammonia-N, mg/L



T = true concentration of ammonia-N in the QL, mg/L

If the QL recovery exceeds the limits of 50 – 150%, the analysis shall be terminated. The cause of the poor recovery must be determined and the problem corrected. The instrument must be re-calibrated and all the samples analyzed after the out-of-control QL standard must be reanalyzed. If, after recalibration, the QL recovery still exceeds the 50-150% limits, the calibration standards must be re-prepared and the instrument recalibrated.

9.3.5 LRB – The laboratory must analyze at least one LRB daily or with each batch of 20 or fewer samples of the same matrix, whichever is more frequent. LRB data are used to assess contamination in the laboratory environment. LRB values that exceed the MDL indicate potential laboratory contamination. If the potential contamination significantly impacts the analytical results, the LRB must be re-prepared along with affected samples, and reanalyzed.

9.3.6 LFB – A LFB must be prepared and analyzed with each batch of 20 or fewer samples. The LFB assures that the calibration standards used to calibrate are accurate. The LFB is the QCS. The recovery of ammonia in the LFB is calculated as follows:

$$\%R = \frac{\text{LFB}}{s} \times 100$$

Where

%R = percent recovery

LFB = measured concentration of ammonia-N in the LFB, mg/L

s = ammonia-N concentration in the LFB, mg/L

The recovery of ammonia in the LFB must be within the 90 – 110% limits. If the recovery exceeds the limits, the analysis system is judged to be out-of-control, and the source of the problem must be identified and resolved before continuing analyses.

9.3.7 LD – Sample homogeneity can affect the quality and interpretation of the data. LD results can be used to assess sample homogeneity.

One LD must be prepared for every 10 routine samples of the same matrix in a sample batch (e.g., 1 LD for a batch containing 1-10 routine samples, 2 LDs for a batch containing 20 routine samples, etc.). Shake the sample selected as the LD, obtain a representative aliquot, and proceed with the sample preparation and analysis, treating the LD sample as a routine sample.

Calculate the relative percent difference (RPD) using the following equation:



$$RPD = \frac{(C_{ld} - C)}{(C_{ld} + C) / 2} \times 100$$

Where

RPD = relative percent difference
 C_{ld} = measured ammonia-N in the LD, mg/L
C = measured ammonia-N in the routine sample, mg/L

The relative percent difference (RPD) must be $\leq 20\%$ for samples with ammonia levels greater than or equal to 5X the QL. For other samples, the absolute difference between duplicate results must be less than the QL. If the control limits are exceeded, flag all associated analyte results. Document actions in the **Notes** section of the LIMS analytical results report.

9.3.8 LFM – The LFM is designed to provide information about the effect of sample matrix on the measurement system. One LFM must be prepared for every ten routine samples of the same matrix in a sample batch. The sample chosen as the LD should be used as the sample LFM. Samples identified as field blanks cannot be used for LFM sample analysis. The analyte concentration must be high enough to be detected above the original sample and should not be less than 4X the MDL. Percent recovery may be calculated using the following equation:

$$\%R = \frac{C_{lfm} - C}{s} \times 100$$

Where

%R = percent recovery
 C_{lfm} = measured concentration of ammonia-N in the LFM, mg/L
C = measured concentration of ammonia-N in the routine sample, mg/L
s = expected ammonia-N concentration in the LFM, mg/L

If the value of C is less than 4X the value of s, the acceptance window for %R is 75 – 125%. If the recovery falls outside the acceptance window other QC data must be examined to determine if a matrix problem exists. If the laboratory performance for that analyte is in control (i.e., the CV, QL, and the LFB results are acceptable, the poor LFM recovery is most likely matrix related. Lab duplicate results should also be examined to gain additional insight as to whether the matrix components or matrix heterogeneity are the cause of the unacceptable recovery. In either case, the problem should be discussed in the report and the data user informed that the result for that analyte in the unfortified sample is suspect due either to heterogeneous nature of the



sample or a matrix effect. Flag any out-of-control analytes. Document actions in the **Notes** section of the LIMS analytical results report.

10. ANALYTICAL PROCEDURES

10.1 CALIBRATION AND STANDARDIZATION – Ammonia is determined colorimetrically using the Lachat Automated Ion Analyzer. The analyst is advised to follow the recommended operating conditions provided by the manufacturer. It is the responsibility of the analyst to verify that the instrument configuration and operating conditions satisfy the analytical requirements, to maintain quality control data verifying instrument performance.

10.1.1 Instrument Set-up

- 1) Turn on the Lachat QuikChem 8000 FIA+ instrument and allow the colorimeter to warm up for about 30 minutes.
- 2) Set up the ammonia manifold and allow the heater to warm up to 60°C.
- 3) Turn on the pump and set the speed to 35 RPM
- 4) Download the ammonia method in the computer.
- 5) Pump reagent water through all reagent lines and check for leaks and smooth flow. Switch to reagents and allow system to equilibrate until a stable baseline is achieved. Don't forget to place the waste lines into the ammonia analysis waste container.

10.1.2 Calibration and Sample Analysis

- 1) Pour the five calibration standards and the blank into standard tubes and position them in decreasing order in the standards rack at the rear of the autosampler.
- 2) Load the analytical and QC samples into the samples rack using the sample tubes.
- 3) The usual sample loading sequence is listed in the following table:

Row	Sample ID	Cup #	Sample Type	Level
1	Cal Std 1	1	Cal Std	1
2	Cal Std 2	2	Cal Std	2
3	Cal Std 3	3	Cal Std	3



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4	Cal Std 4	4	Cal Std	4
5	Cal Std 5	5	Cal Std	5
6	Blank	6	Cal Std	6
7	ICV	1	Unknown	0
8	ICB	2	Unknown	0
9	QL	3	Unknown	0
10	LFB	4	Unknown	0
11	LRB	5	Unknown	0
12	Sample 1	6	Unknown	0
13	Sample 1 – LD	7	Unknown	0
14	Sample 1 – LFM	8	Unknown	0
15	Sample 2	9	Unknown	0
16	Sample 3	10	Unknown	0
17	Sample 4	11	Unknown	0
18	Sample 5	12	Unknown	0
19	CCV	13	Unknown	0
20	CCB	14	Unknown	0
21	Sample 6	15	Unknown	0
22	Sample 7	16	Unknown	0
23	Sample 8	17	Unknown	0
24	Sample 9	18	Unknown	0
25	Sample 10	19	Unknown	0
26	Sample 11	20	Unknown	0
27	Sample 11 – LD	21	Unknown	0
28	Sample 11 – LFM	22	Unknown	0
29	Sample 12	23	Unknown	0
30	Sample 13	24	Unknown	0
31	CCV	25	Unknown	0
32	CCB	26	Unknown	0
33	Sample 14	27	Unknown	0
34	Sample 15	28	Unknown	0
35	Sample 16	29	Unknown	0
36	Sample 17	30	Unknown	0
37	Sample 18	31	Unknown	0
38	Sample 19	32	Unknown	0
39	Sample 20	33	Unknown	0
40	CCV	34	Unknown	0
41	CCB	35	Unknown	0

- 3) Input the information required by the data system such as concentrations, replicates and quality control scheme.
- 4) Calibrate the instrument by injecting the calibration standards. The system will analyze the calibration standards and calculate a calibration curve prior to



analyzing any of the samples. A correlation coefficient of ≥ 0.995 is the requirement for the calibration to pass. The system will now automatically analyze the samples loaded in the sample tray.

10.1.3 Post-analysis Review

- 1) QC Sample Results – Review the results for all QC samples for compliance with the criteria specified in Section 9. If results are not acceptable, take appropriate corrective action.
- 2) Off-scale Results – Review results for samples that exceed the calibration range. Samples having ammonia concentrations larger than the highest calibration standard must be diluted and reanalyzed.

10.1.4 Instrument Shutdown

- 1) At the end of the run with the pump still turned on, place all reagent lines in reagent water and pump for at least 15 minutes.
- 2) After the 15 minute period, remove the reagent lines from the reagent water and allow the reagent lines to be purged of the reagent water. Observe the tubing on the manifold – when no liquid is apparent in the tubing the pump can be turned off. Cap all reagents, discard all samples and standards into the appropriate waste containers and turn the power off.

10.2 Data Reduction and Reporting – After manual set-up and calibration the software reports results for the analyzed solution in units of mg/L. No further calculations are necessary and values may be reported directly from the data system. All results should be reported using no more than three significant figures; however, no values of less significance than the MDL may be reported. Results less than the MDL should be reported as <MDL value. Values between MDL and the QL will be flagged as estimated (J flag).

10.2.1 Sample results are entered into the Laboratory Information Management System (LIMS) and analytical results are reported.

10.2.2 Before releasing the results, the laboratory conducts data verification and validation. This is done through peer review of the data and validation by another laboratory chemist. The QA Manager makes the final audit and validation prior to the release of the results.



11 DOCUMENTATION

- 11.1 When samples are received, the laboratory personnel verify that the chain of custody is properly filled out. Laboratory personnel may then receive and sign the chain of custody. A copy of the chain of custody is included in the data package (Attachment E).
- 11.2 Each standard and reagent prepared for the analysis is entered in the Inorganic Standard Preparation Logbook (Attachment F) and Inorganic Reagent Preparation Logbook (Attachment G) respectively. Copies of the appropriate page(s) is (are) included in the data package.
- 11.3 The Omnion FIA Software Report that contains the operator's/analyst's name, calibration and QC data, $\text{NH}_3\text{-N}$ results in mg/L, sample analysis date and time, client sample IDs/station locations, must be included in the data package (Attachment C).
- 11.4 A QC Summary Report that contains the QC sample results and evaluations must be included in the data package (Attachment D).
- 11.5 The LIMS Analytical Results Report that contains the analyst's name, analytical method, $\text{NH}_3\text{-N}$ results in mg/L, reporting limit, sample analysis date and time, GEPA Lab Sample IDs and client sample IDs/station locations, must be included in the data package (Attachment B).
- 11.6 The data package consists of the following:
- Attachment A: Deviations from Reference Method
 - Attachment B: Analytical Results Report (LIMS Report or Spreadsheet)
 - Attachment C: Omnion FIA Software Report
 - Attachment D: QC Summary Report
 - Attachment E: Chain of Custody
 - Attachment F: Inorganic Standard Preparation Logbook
 - Attachment G: Inorganic Reagent Preparation Logbook

12 REFERENCES

- 12.1 EPA Method 350.1, Determination of Ammonia Nitrogen by Semi-Automated Colorimetry, Revision 2.0, August 1993.
- 12.2 Method 4500- NH_3 H. Automated Phenate Method, Standard Methods For Examination of Water and Wastewater, 20th Edition, 1998.
- 12.3 QuikChem Method 31-107-06-1-B, Determination of Ammonia in Brackish or Seawater By Flow Injection Analysis, Lachat Instruments, January, 2002.



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Attachment A

Deviations From Reference Method

- A.1 This SOP includes routine quality control procedures based on USEPA Region 9 Lab SOP #590 which are in addition to those listed in EPA Method 350.1. These include the analysis of a QL Standard and laboratory duplicate.



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Attachment B: Analytical Results Report
(LIMS Report or Spreadsheet)



- Guam Environmental Protection Agency -

EMAS Laboratory

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Barrigada, Guam 96921

TEL (671) 475 - 1658, FAX (671) 477 - 9402

Date of Report: 08-May-07

To: GEPA - EMAS Assessment Program (EMAP)
Jesse Cruz

Project: GWSA

Attached are the analytical results for sample(s) listed below.

Lab Sample Number	Field Sample ID	Station Location
01974-005	GWSA05-16	Ylig River

If you have any questions regarding this report, please contact the Guam EPA EMAS Administrator. When making inquiries, please reference the Lab Sample Number which appears in the upper right corner of each report page.

A handwritten signature in black ink, appearing to be "MMA", written over a horizontal line.

Analyst

A large, stylized handwritten signature in black ink, written over a horizontal line.

EMAS Administrator

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- Guam Environmental Protection Agency -
EMAS Laboratory

Date of Report: 08-May-07

Field ID: **GWSA05-16**
Submitter: **GEPA - EMAS Assessment Program (EMAP)**
Type of Sample: **Water**

Lab Sample Number: **01974-005**
Date Sample Collected: **16-Apr-07**
Date Sample Received: **19-Apr-07**

Parameter Name	Analytical Method	Date Analyzed	Analyst	MDL	Result	Units
Conductivity	(Dilution Factor: 1)					
Conductivity	EPA 120.1	4/16/2007	eyanit		504	mg/L
Total Suspended Solids (103-105 C)	(Dilution Factor: 1)					
Total Suspended Solids (103-105 C)	SM 2540D	4/17/2007	RP	10	<10	mg/L
Total Dissolved Solids	(Dilution Factor: 1)					
Total Dissolved Solids	SM2540C	4/17/2007	RP	10.00	291	mg/L
Nitrate (as N)	(Dilution Factor: 1)					
Nitrate (as N)	EPA 353.2	4/17/2007	eyanit	0.050	0.058 J	mg/L
Nitrite (as N)	(Dilution Factor: 1)					
Nitrite (as N)	EPA 353.2	4/17/2007	eyanit	0.01	<0.01	mg/L
Ammonia	(Dilution Factor: 1)					
Ammonia	EPA 350.1	5/8/2007	RP/EY	0.01	0.017 J	mg/L
Orthophosphate as P	(Dilution Factor: 1)					
Orthophosphate (as P)	EPA 365.1	4/17/2007	eyanit	0.005	0.021 J	mg/L
Turbidity, NTU	(Dilution Factor: 1)					
Turbidity, NTU	EPA 180.1	4/16/2007	eyanit	0.05	1.30	NTU
pH	(Dilution Factor: 1)					
pH	SM 4500H	4/16/2007	eyanit		7.72	std units

Lab Qualifiers: J = Value is greater than or equal to the method detection limit (MDL) but less than the practical quantitation limit (PQL).

ANALYTICAL REPORT

Analyst: E. Yanit *EY*

GEPA Lab ID	Field Sample ID	Result	Units	MDL	PQL	Analysis Date	Remarks
02285-007	GU10-0051	< 0.010	mg/L	0.010	0.050	6/4/2010	
02285-008	GU10-0052	< 0.010	mg/L	0.010	0.050	6/4/2010	
02285-009	GU10-0053	< 0.010	mg/L	0.010	0.050	6/4/2010	
02286-006	GU10-0009	< 0.010	mg/L	0.010	0.050	6/4/2010	
02286-007	GU10-0011	< 0.010	mg/L	0.010	0.050	6/4/2010	
02288-008	GU10-0001	0.061	mg/L	0.010	0.050	6/4/2010	
02288-009	GU10-0004	< 0.010	mg/L	0.010	0.050	6/4/2010	
02288-010	GU10-0002	0.022 ^J	mg/L	0.010	0.050	6/4/2010	
02288-011	GU10-0003	< 0.010	mg/L	0.010	0.050	6/4/2010	
02288-012	GU10-0005	< 0.010	mg/L	0.010	0.050	6/4/2010	

NOTE: J - Sample result is above the MDL but below the PQL.

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Attachment C: Omnion FIA Software Report

Stock Standard: 1000 mg/L Ammonia as N (ERA Lot # 04086)

OPERATOR: Rudy Paulino
ACQ. TIME: May 8, 2007 13:34:02
DATA FILENAME: C:\OMNION\VEYANIT\050807A.FDT
METHOD FILENAME: C:\OMNION\METHODS\4WS07A.MET
TRAY FILENAME: C:\OMNION\VEYANIT\050807A.TRA

TRAY DESCRIPTION:

Created: May 8, 2007 10:43:29
Modified: May 8, 2007 10:45:14
2007 Wadeable stream samples for Ammonia for samples 01974,01977, 01978

DATA DESCRIPTION:

Created: May 8, 2007 13:34:02
Modified: May 8, 2007 13:34:02
2007Wadeable Stream Samples 01974,01977,01978

Multi-Channel Table
Type: Unknowns
Channel Range: 1 to 8 -- Cup Range: 1 to 50

Cup	Sample ID	Sampling Date	Sampling Time	# of Reps	Ammonia (ppm)	Man Dil Factor	Auto Dil Factor
1	ICV (0.5 ppm)	08 May 2007	13:41:13	1	0.508	1.0	1.00
2	ICB	08 May 2007	13:42:12	1	-0.003	1.0	1.00
3	QL 0.05ppm	08 May 2007	13:43:10	1	0.046	1.0	1.00
4	LRB	08 May 2007	13:44:08	1	-0.003	1.0	1.00
5	LFB (ERA 10095) 0.5 ppm	08 May 2007	13:45:07	1	0.454	1.0	1.00
6	01974-05	08 May 2007	13:46:04	1	0.017	1.0	1.00
7	01974-05 LD	08 May 2007	13:47:01	1	0.014	1.0	1.00
8	01974-05 LFM(0.5ppm)	08 May 2007	13:47:59	1	0.526	2.0	1.00
9	01977-05	08 May 2007	13:48:56	1	-0.004	1.0	1.00
10	01978-05	08 May 2007	13:49:53	1	-0.007	1.0	1.00
11	CCV (0.5 ppm)	08 May 2007	13:50:50	1	0.487	1.0	1.00
12	CCB	08 May 2007	13:51:47	1	-0.003	1.0	1.00
12	Rinse	08 May 2007	13:52:43	2	-0.003	1.0	1.00

Method - Ch. 3 (Ammonia)

METHOD DESCRIPTION:

Created: May 8, 2007 10:42:18
Modified: May 8, 2007 13:34:02
2007 Wadeable stream Ammonia samples 01974,01977, 01978

ANALYTE DATA:

Analyte Name: Ammonia
Concentration Units: ppm
Chemistry: Brackish
Inject to Peak Start (s): 10.0
Peak Base Width (s): 20.000
% Width Tolerance: 100.000
Threshold: 250.000
Inject to BW Baseline Start: 11.695
Inject to BW Baseline End: 76.822
Inject to BW Integ Start: 27.279
Inject to BW Integ End: 72.868
Autodilution Trigger: On
QuikChem Method:

CALIBRATION DATA:

Levels:
1 : 1.000 2 : 0.500 3 : 0.250 4 : 0.100
5 : 0.050 6 : 0.000
Calibration Rep Handling: Replace
Calibration Fit Type: 1st Order Poly
Force Though Zero: No
Weighting Method: None
Concentration Scaling: None

SAMPLER TIMING:

Method Cycle Period: 60.0
Min. Probe in Wash Period: 15.0
Probe in Sample Period: 30.0

*** Prep Sequence Not Enabled ***

VALVE TIMING:

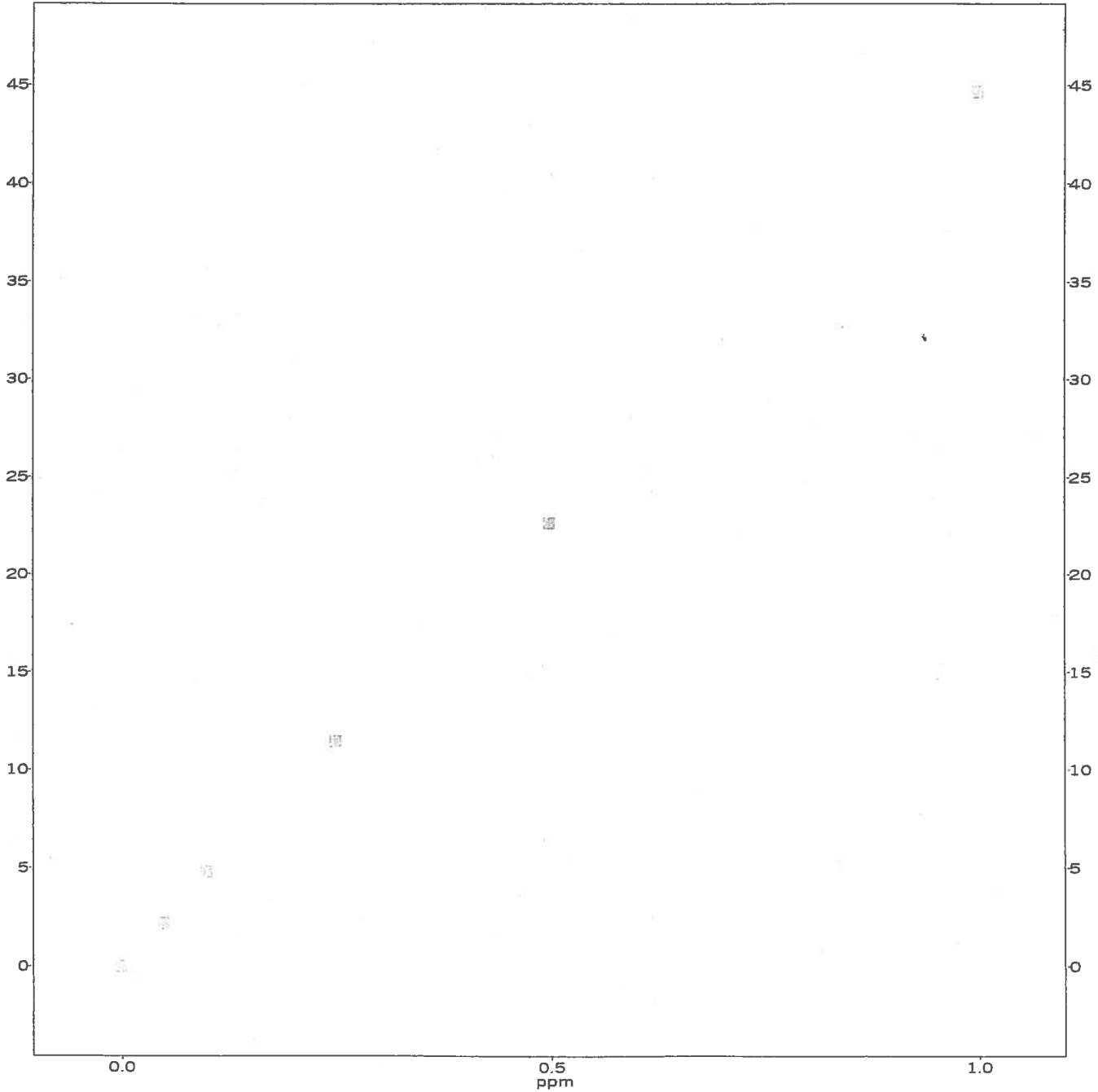
Method Cycle Period: 60.0
Sample Reaches 1st Valve: 18.0
Valve: On
Load Time: 7.0
Load Period: 30.0
Inject Period: 30.0

Ammonia

Level	Area	ppm	Rep 1	Rep 2	Rep 3	Rep 4	Rep 5	Replic STD	Replic % RSD	Residual 1st Poly
1	44682512	1.00	44682512					0.0	0.0	0.3
2	22656238	0.50	22656238					0.0	0.0	-0.7
3	11489592	0.25	11489592					0.0	0.0	-1.4
4	4812883	0.10	4812883					0.0	0.0	-4.0
5	2194117	0.05	2194117					0.0	0.0	9.3
6	5442	0.00	5442					0.0	0.0	

1st Order Poly
 Conc = 2.240e-008 Area - 3.773e-003
 r = 0.9999

Scaling: None - Weighting: None





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Attachment D: QC Summary Report

GUAM EPA LABORATORY

QC Summary Report

Ammonia Determination

GEPA Lab Sample Number:		Sample #		01979, 01977, 01978		Analyst:		RP/ey		Date:		5/8/07	
QC Sample	Calculation	Result	Acceptable (Y/N)	Acceptable Range	Notes								
ICV	$\%R = (\text{measured analyte conc.} / \text{true analyte conc.}) \times 100$ $\frac{0.5077}{0.5} \times 100$	101.5%	Y	90-110%									
ICB		-0.0032	Y	<QL	QL = 0.05 mg/L								
QL	$\%R = (\text{measured analyte conc.} / \text{true analyte conc.}) \times 100$ $\frac{0.0464}{0.05} \times 100$	92.8%	Y	50 - 150%									
LRB		-0.0033	Y	<MDL	MDL = 0.01 mg/L								
LFB (or QCS)	$\%R = (\text{measured analyte conc.} / \text{true analyte conc.}) \times 100$ $\frac{0.453}{0.5} \times 100$	90.8%	Y	90 - 110%									
LD	RPD = $[(\text{meas. analyte conc. in LD} - \text{meas. analyte conc. in routine sample}) / (\text{mean of LD and routine sample conc.})] \times 100$ $\frac{0.0169 - 0.0142}{0.0155} \times 100 = 17.5$ result < QL RPD n/a	result < QL RPD n/a	Y	≤ 20% for samples with analyte levels ≥ 5X QL	For other samples, the absolute difference between duplicate results must be <QL. For samples <QL, RPD is not applicable.								
LFM	$\%R = [(\text{meas. analyte conc in LFM} - \text{meas. analyte conc. in routine}) / (\text{expected analyte conc. of added spike in LFM})] \times 100$ $\frac{(0.526 - 0.017)}{0.5} \times 100$	101.8%	Y	If the measured analyte conc in routine sample is <4X the analyte conc of added spike in LFM, %R is 75-125%	For other samples, %R is 90 - 110%								
CCV	$\frac{0.487}{0.5} \times 100$	97.4%	Y	90 - 110%	Calculation is same as ICV's.								
CCB		-0.003		<QL									
Comments:													



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Attachment E: Chain of Custody

PROJ. NO. GWSADS	PROJECT NAME 2005 Guam Wadable Streams Assessment	SAMPLERS: (Signature)				NO. OF CONTAINERS		REMARKS	
FIELD SAMPLE ID	DATE	TIME	COMP	GRAB	STATION LOCATION				
GWSADS-016	4/16/07	0905		X	41605 Ave Fallet Yalg River				
						NO ₃ , NO ₂ , OP ₄ (0.4 u.f.H.)			
						NH ₄ (0.4 u.f.H.)			
						TDS + TSS			
						pH (conductivity, temperature, etc.)			
						E. coli			
Relinquished by: (Signature)						Date / Time	Received by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)						Date / Time	Received by: (Signature)	Date / Time	Received by: (Signature)
Relinquished by: (Signature)						Date / Time	Received by: (Signature)	Remarks	



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Attachment F: Inorganic Standards Preparation Logbook

GUAM ENVIRONMENTAL PROTECTION AGENCY - EMAS Analytical Program

Inorganic Standard Preparation Logbook

GEPA Standard ID	Standard Description	GEPA Std Receipt Number	Initial Conc. (mg/L, ug/L)	Volume Used (mL, uL)	Final Volume (L, mL)	Final Conc. (mg/L, ug/L)	Preservative	Prep Date	Exp Date	Prep By (Initials)	Remarks
STD041707-09	0.5 ppm PIVL	STD06-007	1000 ppm	25 uL	50 mL	0.5 ppm	✓	4/17/07	4/18/07	EY	2 nd Source
-10	1 ppm NO ₂	STD06-001	1000 ppm	100 uL	100 mL	1 ppm	✓	4/17/07	4/18/07	EY	Calibration
-11	0.5			100 uL	200 mL	0.5 ppm	✓				Spks.
-12	0.05			10 uL	200 mL	0.05 ppm	✓				
-13	0.5	STD06-009	1000 ppm	100 uL	200 mL	0.5 ppm	✓			EY	EY 4/17/07
-13	0.25 ppm NO ₂	STD06-009	1000 ppm	50 uL	200 mL	0.25 ppm	✓	4/17/07	4/18/07	EY	2 nd Source
STD050107-01	5 ppm	STD06-002	1000 ppm	500 uL	100 mL	5 ppm	5 ppm	5/1/07	5/2/07	PPS	
-02	2.5			125 uL	50 mL	2.5 ppm	2.5 ppm				
-03	0.25			25 uL	100 mL	0.25 ppm	0.25 ppm				
-04	2.5 ppm	STD06-013	1000 ppm	625 uL	25 mL	2.5 ppm	2.5 ppm			PPS	2 nd Source
STD050107-05	1 ppm PO ₄	STD06-003	1000 ppm	100 uL	100 mL	1 ppm	✓				
-06	0.5			80 uL	100 mL	0.5 ppm	✓				

GUAM ENVIRONMENTAL PROTECTION AGENCY - EMAS Analytical Program

Inorganic Standard Preparation Logbook

GEPA Standard ID	Standard Description	GEPA Std Receipt Number	Initial Conc. (mg/L, ug/L)	Volume Used (mL, uL)	Final Volume (L, mL)	Final Conc. (mg/L, ug/L)	Preservative	Prep Date	Exp Date	Prep By (Initials)	Remarks
STD050107-07	0.05 P ₀₄	STD06-003	1000 ppm	10 uL	200 mL	0.05 ppm	✓	5/1/02	5/2/12	JPSP	
-08	0.025 P ₀₄	↓	↓	12.5 uL	500 mL	0.025 ppm	✓	↓	↓	JPSP	
STD050807-01	1 ppm NH ₄ -N	STD06-005	1000 ppm	100 uL	100 mL	1 ppm	✓	5/8/12	5/9/12	JPSP	
-02	0.5 ppm NH ₄ -N	↓	↓	50 uL	100 mL	0.5 ppm	✓	↓	↓	↓	
-03	0.25 ppm NH ₄ -N	↓	↓	25 uL	100 mL	0.25 ppm	✓	↓	↓	↓	
-04	0.1 ⁰ ppm NH ₄ -N	↓	↓	10 uL	100 mL	0.1 ppm	✓	↓	↓	↓	
-05	0.05 ppm NH ₄ -N	↓	↓	10 uL	200 mL	0.05 ppm	✓	↓	↓	↓	
-06	0.5 ppm NH ₄ -N	STD04-008	↓	25 uL	50 mL	0.5 ppm	✓	↓	↓	↓	Second Source



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Attachment G: Inorganic Reagent Preparation Logbook

GUAM ENVIRONMENTAL PROTECTION AGENCY - EMAS Analytical Program

Inorganic Reagent Preparation Logbook

GEPA Reagent ID	Reagent Description	Reagent 1 (wt or vol used)	Reagent 2 (wt or vol used)	Reagent 3 (wt or vol used)	Final Volume of H2O Used	Preservative (wt, vol)	Prep Date	Exp Date	Prep By (Initials)	Remarks
RGTD031607-02	Sodium Nitrogenmide	0.875g Solid Nitrogenmide	—	—	250	—	3/16/07	3/23/07	MSR	1-2 weeks
RGTD031507-03	Phenolate Rgt	20.75g Phenol Crystals	8g NaOH	—	250	—	3/16/07	3/24/07	MSR	3-5 days
RGTD031607-04	Sodium Hydroxide	100 NaOH	—	—	200	—	3/16/07	3/17/07	MSR	Daily
RGTD032207-01	Methylphosphate Color Rgt.	17.5ml conc. H2SO4	100.5ml Ammonia Methylphosphate	3.6ml Ant. Pst. Preservative	500	—	3/22/07	3/27/07	EY	
RGTD032207-02	Hydrobromic Acid	12g Oxalic Acid	—	—	200ml	—	3/27/07	3/28/07	EY	
RGTD040907-01	Sodium Nitrogenmide	0.875g Solid Nitrogenmide	—	—	250	—	4/9/07	4/16/07	EY	
RGTD040907-02	Phenolate Rgt.	20.75g Phenol Crystals	8g NaOH	—	250	—	—	4/14/07	EY	
RGTD040907-03	Sodium Hydroxide	100ml NaOH	—	—	200	—	—	4/10/07	EY	
RGTD041707-01	Sulfuric Acid Color Rgt.	10g Sulfuric Acid Crystals	0.05g NED	75ml H2PO4	750	—	4/17/07	5/17/07	EY	
RGTD041707-02	Ascorbic Acid	170g Ascorbic Acid Crystals	2g EDTA	15 N NaOH until PA85 is reabsorbed.	2L	—	4/17/07	4/17/08	EY	
RGTD041707-03	Ascorbic Acid	170g Ascorbic Acid Crystals	—	—	200ml	—	4/17/07	4/24/07	EY	
RGTD051107-01	Ascorbic Acid	15g AA	—	—	250ml	—	5/1/07	5/2/07	MSR	

Approved By: _____
Date Approved: _____

Inorganic Reagent Preparation Logbook

[illegible]

